Particle Metrology and Nanoassembly

As technology migrates toward smaller physical dimensions, new analytical approaches are required to characterize material properties and to investigate critical issues. Our primary focus is the application of metrology and the development of new methods and standards for measuring the physical and surface properties of nanostructured particle systems. Applications include functional materials and devices for catalysis, power generation, and microelectronic, pharmaceutical, and biotechnology industries.

Vince Hackley

Nanocrystalline oxides of alkaline-earth cations produced by a supercritical drying process exhibit unique and highly reactive surface chemistries. As a result, these materials have been studied extensively as destructive adsorbents, catalysts, and bioactive agents. Investigations were concluded regarding the role of cation size on the evolution of microstructure in these materials and on the dispersion properties in aqueous NaCl solution. A series of small-angle neutron scattering (SANS) experiments were performed at the NIST reactor in collaboration with researchers at Kansas State University. These results indicate a complex picture for structure formation during the drying and annealing processes, with string-like gel morphology giving way to fractally rough particulate assemblies of compacted nanocrystals.

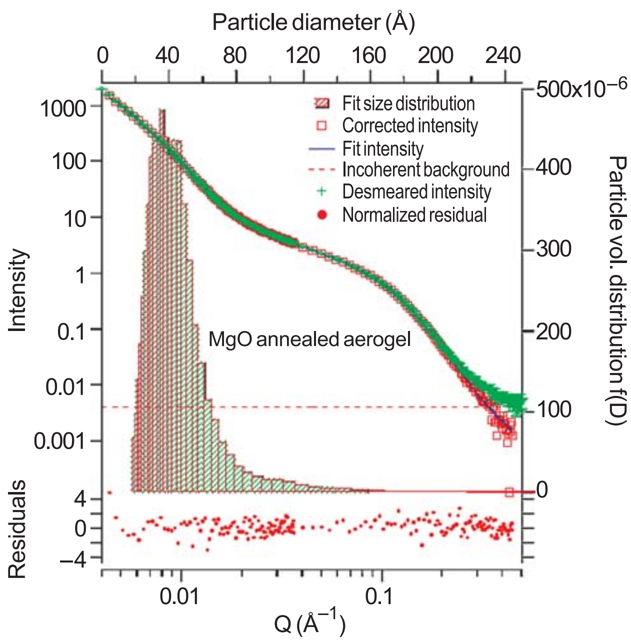

***Figure 1:** Maximum entropy fit to SANS data for annealed MgO aerogel.*

Data were analyzed using the Unified Model of Beaucage and the maximum entropy method (see Figure 1). The scale of the finest structural features increases with increasing cation size for the annealed product. Analysis indicates the absence of mass fractal structures but the presence of surface fractal-like objects. A broad correlation peak in the data for heat-treated Mg and Ca oxides gives evidence for some local ordering of the nanocrystals. This data will help provide a more complete understanding of the structural development in this complex and technologically important system.

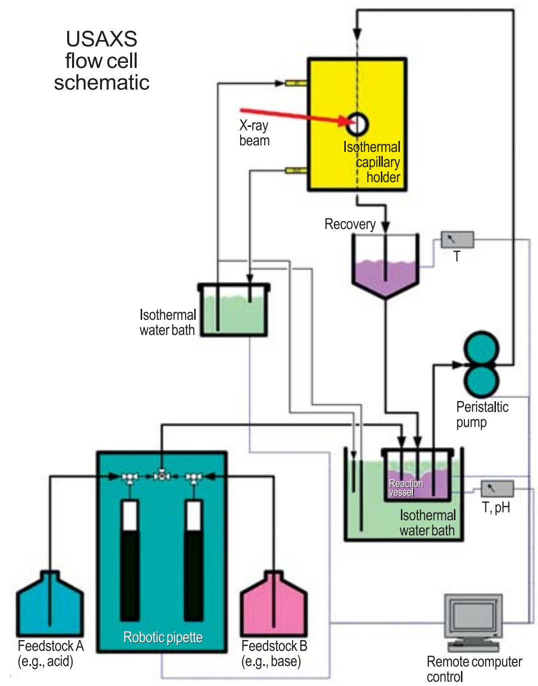

***Figure 2:** Schematic diagram of capillary flow cell.*

A capillary flow-cell (Figure 2) was developed and commissioned for the ultra-small-angle x-ray scattering (USAXS) instrument on the UNICAT beam line at the Advanced Photon Source. This new capability will permit *in situ* investigations of complex multiphase particulate systems under controlled flow conditions. Initial applications include the dispersion of single-wall carbon nanotubes in collaboration with Rice University and depletion effects in binary colloidal suspensions.

Contributors and Collaborators

A. Allen, L. Lum (Ceramics Division, NIST); D. Ho (NIST Center for Neutron Research); K. Klabunde (Kansas State University); M. Pasquali (Rice University); P. Jemian (University of Illinois)